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METHODS OF CHEMICAL ANALYSIS OF SILVER-MANGANESE BRAZING ALLOYS

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METHODS OF CHEMICAL ANALYSIS OF SILVER-MANGANESE BRAZING ALLOYS

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF SILVER-MANGANESE BRAZING ALLOYS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 15 July 1968, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 For correct analysis of silver-manganese (heat-resistant) brazing alloys, methods of chemical analysis have been laid down in the standard.

0.3 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes methods for determining silver and manganese content in silver-manganese (heat-resistant) brazing alloys specified in IS : 2927-1964†.

2. SAMPLING

2.1 Laboratory Sample — It shall be drawn and prepared in accordance with IS : 1817-1961‡.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960§) shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Rules for rounding off numerical values (*revised*).

†Specification for brazing alloys.

‡Methods of sampling of non-ferrous metals for chemical analysis.

§Specification for water, distilled quality (*revised*).

4. DETERMINATION OF SILVER BY THE GRAVIMETRIC METHOD

4.1 Outline of the Method — The sample is dissolved in nitric acid [illegible] silver precipitated as silver chloride by means of hydrochloric [illegible] The precipitate is filtered, washed, dried and weighed.

4.2 Reagents

4.2.1 *Dilute Nitric Acid* — 1 : 1, 3 : 7 and 3 : 97 (*v/v*).

4.2.2 *Sodium Bismuthate* — solid.

4.2.3 *Sulphurous Acid Solution* — saturated.

4.2.4 *Dilute Hydrochloric Acid* — 1 : 9 (*v/v*).

4.3 Procedure

4.3.1 Transfer one gram of an accurately weighed sample to 250-ml beaker. Add 20 ml of dilute nitric acid (1 : 1) and leave on hot-plate. Decant the solution if there is any residue, bring it into solution by treatment with dilute nitric acid (3 : 7) and a pinch of sodium bismuthate followed by addition of a few drops of sulphurous acid solution. Mix with the decanted solution and transfer to a 200-ml volumetric flask and make up the volume.

4.3.2 Transfer 50 ml to a 250-ml beaker. Add 25 ml of dilute hydrochloric acid slowly with constant stirring. Let it stand for about an hour.

4.3.3 Decant the solution through a weighed sintered glass crucible. Wash the precipitate with warm dilute nitric acid (3 : 97) till free from chloride and decant through the crucible.

4.3.4 Dry the crucible and precipitate at about 110°C. Cool in a desiccator to room temperature and reweigh.

4.4 Calculation

$$\text{Silver, percent} = \frac{A}{B} \times 75{\cdot}26$$

where

A = weight in g of silver chloride, and
B = weight in g of the sample represented by aliquot taken.

5. DETERMINATION OF MANGANESE BY THE BISMUTHATE (VOLUMETRIC) METHOD

5.1 Outline of the Method — Manganese in the solution of the sample is oxidized to permanganic acid by means of sodium bismuthate in nitric acid medium, and the permanganate thus formed is reduced by a measured volume of standard ferrous ammonium sulphate solution, excess of which is titrated with standard potassium permanganate solution.

5.2 Reagents

5.2.1 *Dilute Nitric Acid* — 1 : 3 and 5 : 95 (*v*/*v*).

5.2.2 *Sodium Bismuthate* — solid.

5.2.3 *Standard Ferrous Ammonium Sulphate Solution* (0·1 N) — Dissolve 39·2 g of ferrous ammonium sulphate crystals $[Fe (NH_4)_2 (SO_4)_2, 6H_2O]$ in cold dilute sulphuric acid (5 : 95) and make up to one litre with this in a volumetric flask. Standardize against standard potassium permanganate solution (*see* **5.2.5**).

5.2.4 *Phosphoric Acid* — 85 percent.

5.2.5 *Standard Potassium Permanganate Solution* (0·1 N) — Dissolve 3·2 g of potassium permanganate in one litre of water. Let it stand in the dark for 2 weeks and filter without washing through a Gooch crucible or fritted-glass crucible of fine porosity avoiding contact with rubber or other organic materials. Store in a dark-coloured glass stoppered bottle. Standardize against sodium oxalate (analytical reagent) dried at about 110°C and by weighing 0·134 g of the salt dissolving in 250 ml of dilute sulphuric acid (5 : 95) and titrate against the permanganate at 55° to 60°C to a faint pink end-point. Adjust the strength of the permanganate solution such that 0·134 g of sodium oxalate require exactly 20 ml of the permanganate solution.

5.3 Procedure

5.3.1 Dissolve 0·5 g of an accurately weighed sample in a 500-ml conical flask in 50 ml of dilute nitric acid (1 : 3). In order to destroy the organic matter and to oxidize other reducers, add to the solution, which has been heated to boiling, one gram of sodium bismuthate in small portions. If, during boiling, the pink colour of permanganic acid disappears or the residue of manganese dioxide dissolves, it is necessary to add a further quantity of sodium bismuthate. The formation of permanent precipitate of manganese dioxide indicates complete oxidation of organic and other reducing material. Whilst still boiling add dropwise standard ferrous ammonium sulphate solution until complete solution of the precipitate of manganese dioxide is obtained.

5.3.2 Cool the solution to 10° to 15°C and add 50 ml of cold dilute nitric acid (1 : 3). Add 2·6 g of sodium bismuthate for each 0·1 g of manganese to the cooled solution and stir vigorously for two minutes. Filter immediately through a funnel packed with ignited asbestos pad.

The filtrate should be clear. Wash the filter and the residue with cold dilute freshly boiled nitric acid (5 : 95) until washings are entirely colourless.

5.3.3 Add to the filtrate a measured excess of standard ferrous ammonium sulphate solution, and 5 ml of phosphoric acid. Stir and titrate the excess with standard potassium permanganate solution to the appearance of faint pink colour.

5.3.4 Run a blank through all stages of the procedure using the same amount of ferrous ammonium sulphate solution as added in **5.3.3** and titrate with standard potassium permanganate solution.

5.4 Calculation

$$\text{Manganese, percent} = \frac{(A - B)\,C}{D} \times 1{\cdot}1$$

where

A = volume in ml of standard potassium permanganate solution required to titrate the blank,

B = volume in ml of standard potassium permanganate solution required to titrate the excess ferrous ammonium sulphate solution,

C = normality of the standard potassium permanganate solution, and

D = weight in g of the sample taken.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Definition
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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